41 DETERMINATION OF LEAD CONTENT IN ALCOHOLIC BEVERAGES BY ATOMIC ABSORPTION SPECTROMETRY

41.1 Summary

41.1.1 The determination of lead content by flame atomic absorption spectrometry is used to help identify illegally distilled spirits. Samples are heated to high temperatures (approx 2300° C) in an acetylene/air flame. The flame dissociates chemical bonds and releases free metal atoms which absorb light. The amount of absorbed radiant energy is a function of concentration of lead present in the sample.

41.2 Reagents

41.2.1 95% ethanol (grain alcohol)

41.2.2 Lead oxide, 1000 ppm in 0.3 M nitric acid

41.2.3 Illegally distilled spirit control

41.2.4 Legally distilled spirit control (vodka)

41.3 Solutions and standards

41.3.1 40% ethanol. Pipet 421 mL 95% ethanol into a 1 L volumetric flask and QS to volume with dH_2O.

41.3.2 5.0 ppm lead standard. Pipet 5 mL of 1000 ppm lead standard into a 1 L volumetric flask and QS to volume with 40% ethanol.

41.3.3 0.5 ppm lead standard. Pipet 10 mL of the 5.0 ppm lead standard into a 100 mL volumetric flask and QS to volume with 40% ethanol.

41.3.4 1.0 ppm lead standard. Pipet 20 mL of the 5.0 ppm lead standard into a 100 mL volumetric flask and QS to volume with 40% ethanol.

41.3.5 2.0 ppm lead standard. Pipet 40 mL of the 5.0 ppm lead standard into a 100 mL volumetric flask and QS to volume with 40% ethanol.

41.4 Apparatus

41.4.1 Pipets, Class A, 5 mL, 10 mL and 20 mL volume

41.4.2 Flame Atomic Absorption Spectrometer (Varian SPECTRAA 55B)

41.4.2.1 Parameters. Instument parameters may be adjusted to improve performance.

41.4.2.1.1	Instrument mode:	Absorbance Emission
41.4.2.1.2	Active lamp:	2
41.4.2.1.3	Active current:	8
41.4.2.1.4	Standby current:	0

41.4.2.1.5	D2 correction:	Yes
41.4.2.1.6	Gas type:	Air acetylene
41.4.2.1.7	Wavelength:	Other
41.4.2.1.8	Other:	217 nm
41.4.2.1.9	Slit:	1.0

41.5 Procedure

41.5.1 Ensure AA spectrophotometer exhaust system is on.

41.5.2 Turn on gas supplies in the following order:

41.5.2.1 Air at 50-60 psi

41.5.2.2 Acetylene at 12-15 psi

41.5.3 Turn on AA power. Load method (1 Pb 217).

41.5.4 Ignite flame.

41.5.5 Allow spectrophotometer to warm up for at least 15 minutes.

41.5.6 Aspirate lead free 40% ethanol (blank) and zero instrument.

41.5.7 Aspirate each lead calibrator solution. Read results, calibrate instrument and record absorbance on lead worksheet.

41.5.8 Aspirate samples and read results. The AA will take 3 readings and average the three absorbances and calculate lead concentration (in ppm).

41.5.9 Record the absorbances and concentration (ppm) of each sample on the lead worksheet.

41.5.10 Repeat 41.5.8 and 41.5.8 on case samples.

41.5.11 After all samples have been run, rerun at least one calibrator as a sample and record on worksheet.

41.6 Interpretation

41.6.1 The tolerable lead limit for legally produced beverages is 0.2 ppm. Levels equal to or greater than 0.3 ppm indicate the beverage was illegally distilled.

41.6.2 Lead contents in excess of 3.5 ppm can be hazardous to consumers.

41.7 References

41.7.1 AOAC 14th edition, 25.115 (p 464), 1984

41.7.2 Varian Spectometer AA 50/55 Instrument Manual

41.7.3 The Pharmacological Basis of Therapeutics. Goodman and Gilman. 5th edition, p 939